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PHYSICAL-CHEMICAL NITROGEN REMOVAL FROM MUNICIPAL WASTEWATER

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Robert A. Taft Water Research Center
Cincinnati, Ohio

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Environmental Protection Agency
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ABSTRACT

Selective ion exchange for ammonia removal, using the natural zeolite, clinoptilolite, was applied to lime clarified and filtered raw wastewater both before and after carbon adsorption. Intermittant operation of the ion exchange unit with minimum operator surveillance produced an average of 75% removal of NH_3-N over a 10 month period. With 24 hour surveillance of the operation, and an inlet NH_3-N of 12 mg./l., the residual NH_3-N averaged 0.6 mg./l. for a 95% removal.

Breakpoint chlorination for ammonia removal was applied in the pilot plant to filtered secondary effluent and to selective ion exchange effluent. Chlorination of filtered secondary effluent in an agitated reactor removed essentially all the NH_3-N with 95% conversion to N_2 gas. The $CL:NH_3$-N weight ratio required for successful breakpoint was 8 to 9:1. Base usage, a function of chlorine dosage and the reaction pH, ranged from 0.88 pounds NaOH per pound of CL_2 at pH 6.0 to 1.67 pounds per pound at pH 7.9. Nitrate formation, a function of pH, ranged from 3.6% of influent NH_3-N at pH 6.0 to 5.9% at pH 8.0. Operation at pH greater than 6.0 with good mixing and chlorine dosage control produced essentially no nitrogen trichloride.

Physical-Chemical Nitrogen Removal From Municipal Wastewater*

By

Alan F. Cassel, Thomas A. Pressley, Walter W. Schuk, and Dolloff F. Bishop**

The physical-chemical methods that may be considered for removing ammonia from municipal wastewaters include air stripping, selective ion exchange and breakpoint chlorination. Air stripping has been the subject of several recent papers (1, 2, 3). Unfortunately, ammonia volatility in the air-water system decreases with decreasing temperatures and stripping efficiency decreases sharply for any selected air to liquid rate. Other limitations are cold weather freezing of the towers and calcium carbonate scaling of tower packing.

The selective ion exchange process, with a natural zeolite, clinoptilolite, has been developed by Battelle-Northwest for ammonia removal (4). The clinoptilolite exchanges Na^{+} or Ca^{++} ions for NH_4^{+} ions in the wastewater. Clinoptilolite, obtained from the Baroid Division of National Lead's deposits at Hector, California, is mined as a rock and then crushed and sieved to a 20 x 50 mesh particle size.

The Battelle exchange process is carried out in columns packed with 20 x 50 mesh clinoptilolite. The columns are regenerated by a lime-salt mixture. Concentrated ammonia is removed from the reused regenerant by air

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stripping. Although selective for ammonia, the zeolite also removes cations present in the water. Studies by Battelle-Northwest (5) reveal that with an influent Ca^{++} of 30 mg./l. and NH_3-N of 14 mg./l., half the ion exchange capacity is used by Ca^{++} ions.

Ammonia removal by breakpoint chlorination proceeds according to the overall reaction:

(1) $3Cl_2 + 2NH_3 \longrightarrow N_2 + 6HCl$

The series of steps to oxidize the ammonia to nitrogen gas are:

(2) $Cl_2 + H_2O \longrightarrow HOCl + HCl$

(3) $NH_4^+ + HOCl \longrightarrow NH_2Cl + H_2O + H^+$

(4) $2NH_2Cl + HOCl \longrightarrow N_2 + 3HCl + H_2O$

The "breakpoint" is defined as the point where the NH_3-N is reduced to zero, free available chlorine is detected, and the total residual chlorine is minimized.

Undesirable side reactions may also occur:

(5) $NH_2Cl + HOCl \longrightarrow NHCl_2 + H_2O$

(6) $NHCl_2 + HOCl \longrightarrow NCl_3 + H_2O$

(7) $NH_4^+ + 4Cl_2 + 4H_2O \longrightarrow HNO_3 + H_2O + 8HCL + H^+$

A laboratory study at the EPA-DC Pilot Plant (6) revealed that, with proper mixing and pH control, breakpoint chlorination of ammonia in wastewaters oxidized 95 - 99% of the ammonia to nitrogen gas. When chlorine demands other than ammonia were removed by pretreatment of the wastewater, the chlorine dosages required for breakpoint approached the stoichiometric weight ratio (7.6:1) of Cl:N in Equation 1.

In the study over the pH range 5.0 to 8.0, the chlorine dosage was minimized at pH's between 6.0 - 7.0. The minimum NO_3^- production (1.5% of

the NH_3-N) occurred at pH 5.0. At pH 8.0, the NO_3^- increased to 10% of the influent NH_3-N. The NCl_3 production at the breakpoint decreased from 1.5% of the influent at pH 5 to 0.25% at pH 8.0. Temperature did not affect the product distribution or the required chlorine dose in the range 40-100°F.

This study evaluates the ammonia removal capabilities of the Battelle ion exchange pilot process within a physical-chemical treatment system, pilot scale breakpoint chlorination of filtered secondary effluent, and the combination of ion exchange and chlorination.

Ion Exchange Pilot System

The Battelle pilot plant, housed in a 40-foot trailer, consisted of three exchange columns, a stripping tower, regenerant mixing tank, air blower, and air heater. Figure 1 is a flow diagram showing two ion exchange columns in service and one under regeneration. The 500-gallon fiberglass exchange columns were 39 inches in diameter and eight ft. tall. Each column was packed with approximately 1870 pounds of zeolite and was operated under 35 p.s.i.g. pressure.

The stripping tower was a 43-inch diameter fiberglass tower packed with seven ft. of 1-inch polypropylene Intalox saddles. The tower drained into a 200 gal. agitated tank, where lime was added under pH control. Air for the stripping tower was supplied by the 10-h.p. blower and was heated by a 60-k.w. heater.

A compact piping cross header, installed underneath the trailer, contained 36 electrically-operated ball valves, which permitted the liquid flow to be routed anywhere in the process.

The influent stream, controlled at pH 7.0 - 7.5, was pumped downflow through two clinoptilolite beds in series at loading rates from 1 to 6 gal./

(min.)(sq. ft.). The operation for the three ion exchange columns was:

1. Two beds were in service downflow while the third was being regenerated.
2. After the first bed in service approached maximum ammonia loading (ammonia in effluent was 90% of influent concentration), it was taken from service. The second bed was placed in the number one position and the freshly regenerated bed was placed in the number two position.
3. A column was regenerated by pumping a solution of Na^{+} and Ca^{++} ions (sodium chloride and lime) at a pH of 11.0 upflow through the zeolite.

The regenerant was recirculated through the bed at a loading rate of 7.2 gal./(min.)(sq.ft.) for three hours to build up the ammonia concentration to approximately 500 mg./l. NH_3-N. The regenerant flow was decreased to 3 gal./ (min.)(sq.ft.) and then recycled through both the zeolite bed and the stripper until the NH_3-N was reduced to 20 mg./l. (average time 12 hours). During regeneration, the pH was automatically maintained at 11.

A portion of the regenerant solution which remained in the mixing tank was reused from batch to batch. Following regeneration, the column was backwashed with ammonia free water to remove the lime deposits. The air stripper was operated at a liquid rate of 25 gal./min. with an air to liquid ratio of approximately 100 cu.ft. of air per gal. of water.

The first five months of operation (82 days) were on lime clarified and filtered raw wastewater. In July 1970, the ion exchange unit was placed after carbon adsorption in the physical-chemical system because the anaerobic growths on the carbon were converting organic nitrogen to ammonia. Thus, more ammonia was available for ion exchange on the zeolite after carbon

adsorption. The air stripper was run with heated air for the first month and with ambient air during the remainder of its operation. Regenerant solution was not reused during one month (December) of cold weather operation.

Chlorination Pilot Plant

The chlorination reactor was a 1,200 gal. vessel, four ft. in diameter and twelve ft. tall (Figure 2). The vessel was an existing pH control tank which was larger than needed for chlorination and only a portion of its volume was used. The reaction volume was the bottom three feet of the vessel (310 gal.) which was divided into three equal sections, separated by epoxy-coated plywood inserts. In each section, a propeller mixer mounted on a common shaft, driven by a three h.p. motor, was rotated at 224 r.p.m.

A pump on the effluent stream recycled 11 gal./min. into the first mixing stage. Chlorine was added to the recirculation loop through a Wallace & Tiernan chlorinator and injector nozzle. The dose rate was manually set to provide a dosage equal to an 8.5 - 9-0:1 Cl:N weight ratio. The pH of the water in the reactor was maintained automatically by pumping a 1 - 2% NaOH solution into the line ahead of the chlorine injector. The NaOH feed was controlled by an inline pH probe on the discharge effluent. The chlorinated effluent was then pumped downstream to carbon columns.

A Technicon Auto-Analyzer monitored the NH_3-N concentration of the feed and effluent. The free chlorine in the effluent was titrated manually.

In the study, the average analysis of the feed (filtered secondary effluent) for BOD, COD, TOC, and suspended solids was 16 mg./l.,

41 mg./l., 18 mg./l. and 25 mg./l., respectively. The pH of the feed ranged from 7.0 to 7.3 with an alkalinity of 120 - 150 mg./l. as $CaCO_3$. During all the runs, the water temperature was 58°F. The flow rates ranged from 25 to 35 gal./min. with a residence time in the reaction zone from 6.8 to 8.6 minutes.

During construction of the systems described above, chlorination was also attempted on the ion exchange effluent in the plant using an eductor to mix chlorine water and wastewater in a 2-inch pipe. The NH_3-N ranged from 3.0 to 9.4 mg./l. and the flow rate was 20 gal./min. Samples were taken six feet downstream of the eductor. The ion exchange effluent was also chlorinated in the laboratory with the pilot plant's chlorine water.

Analytical Procedures

Samples around the ion exchange unit were composited every 24 hours and analyzed for TOC, BOD, COD, Total $PO_4^{\equiv}$, TKN, and NH_3-N. Grab samples were analyzed for NH_3-N by the Nessler method (7).

In the laboratory, TOC was measured on a Beckman Carbonaceous Analyzer (8), BOD was determined by the probe method (9), Total $PO_4^{\equiv}$ was determined by the persulfate method (10), and NH_3-N was analyzed on a Technicon Automatic Analyzer (9). All other analyses employed the procedures of Standard Methods (7).

The chlorination study required field measurements for NH_3-N, which were analyzed on a programmed Technicon Automatic Analyzer (9). Free and combined chlorine were measured by the Modified Palin Method (11) with N, N-diethyl-p-phenylenediamine oxalate as the indicator. Nitrogen trichloride was analyzed according to the spectrophotometric procedure of

Czeh et al. (12). Laboratory measurements for nitrate-nitrite (13) were determined on a Technicon Automatic Analyzer.

Ion Exchange Operations

Because of mechanical shutdowns, the unit was operated for 120 days during the ten month test period. The average run lasted 24 hours before switching columns. Ammonia removals (daily composites) for the intermittent operation (Figure 3) averaged 75% with residuals of 3.0 mg./l. NH_3-N. COD and phosphorus were removed from the wastewater by the zeolite bed (Table 1). The suspended solids, however, increased and an increased turbidity appeared in the effluent. The turbidity was produced by lime deposits from poor regenerations.

The large fluctuations in the effluent NH_3 concentrations show the effects of minimum manpower for the operational problem. Electrical valve malfunctions were frequently responsible for mixing of the regenerant and service cycle streams, a major contributor to the poor removal.

Ammonia removals from the D.C. lime clarified raw wastewater (14), however, (2-hour grab samples) averaged 95% during periods of good operation with 24-hour surveillance (Figure 4). The seventh day values were excluded from the calculation of the average ammonia removal because of incomplete regeneration of the replacement column. In the period of efficient operation, the average run lasted 30 hours before regenerating the lead column. Bed loadings ranged from 3.6 to 4.8 gal./(min.)(sq. ft.).

The most important variable for controlling the process was the pH during regeneration. With a low pH (less than 10.5), not all of the ammonia was removed from the bed and made available for air stripping. A high

regenerant pH (greater than 12.0) left deposits of lime on the zeolite, which later, during a service cycle, raised the pH of the feed and converted NH^+_4 to unionized NH_4OH. The pH monitoring of lime solutions required frequent checks of the pH electrodes to prevent their scaling. Thus the majority of the daily operational problems were caused by a malfunctioning pH meter and lime scaling difficulties.

During the first five months of operation, the water contained approximately 18.1 mg./l. of BOD and 45 mg./l. of COD, with potential for biological growth on the zeolite. The pressure drop across the bed increased on every run and the flow had to be reversed for one minute at least twice a run. The high differential pressure, however, was not cumulative from run to run as the high pH regeneration minimized growth.

When ion exchange was placed after carbon adsorption, influent BOD averaged 5.8 mg./l. the COD, 16 mg./l. Microscopic examination of the regenerant solution showed evidence of biological growth occurring during a service cycle. The growth did not increase the pressure drop through the bed. During the early part of regeneration cycles, larger quantities of lime (approximately a 10% increase) were unexpectedly needed to maintain the regenerant solution at pH 11.0. The ammonia removal capabilities of the zeolite were not affected.

After 90 days of operation, the stripping tower packing was plugged with calcium carbonate scale and lime. The lime scale was soft and easily removed. The calcium carbonate scale was hard and had to be removed by agitating the packing in water. The amount of lime found in the stripping tower was caused by the erratic operation of the pH controller. The pH control problem was also responsible for the large quantities of

backwash water (6-8% of total service flow) required after each regeneration.

Chlorination Operations

Chlorination of ion exchange effluent in the eductor mixing system did not achieve breakpoint and excessive NCl_3 formation occurred. The pH decreased from 7.3 to 6.5. A comparative test in the laboratory with good mixing achieved breakpoint between the 8:1 and 9:1 Cl:N weight ratio (Figure 5). The pH decreased from 7.6 before chlorination to 6.1 at the breakpoint. Another plant test with the pH controlled at 7.0 by caustic addition still did not achieve breakpoint. Thus, the eductor did not provide adequate mixing.

Chlorination of filtered secondary effluent in the chlorination reactor achieved breakpoint in the pH range 6.0 to 8.0. Typical breakpoint data at pH 7.0 - 7.5 (Figure 6) revealed the relation between NH_3-N, free chlorine, and total residual chlorine. Breakpoint occurred at a Cl:N weight ratio between 8 and 9:1.

In repeated tests, (Table 2) the influent NH_3-N concentrations, ranging from 12.9 to 21.0 mg./l., were reduced to less than 0.1 mg./l. NH_3-N with Cl:N weight ratios from 8.4 to 9.2:1. The amount of free residual chlorine increased with increasing overdoses of chlorine.

Caustic requirements in the pH range 6.0 to 7.9 (Table 3) increased with increasing reaction pH. Based on Equation 2, the stoichiometric NaOH requirement for maintaining an influent pH was 1.13 pounds NaOH per pound of chlorine added. Calcium hydroxide solution was also used. One run at pH 7.0 indicated approximately 1.0 pound CaO required per pound of Cl_2.

The formation of nitrate was minimized by operating in the lower

pH range (Table 4). In contrast, the formation of nitrogen trichloride decreased with increasing pH. More importantly, NCl_3 concentration increased sharply for chlorine dosage above the breakpoint (Table 5). Thus, good mixing, pH control, and chlorine dosage control were required to minimize undesirable byproducts.

Because NCl_3 formation is more dependent on chlorine dosage, rather than pH, operation at pH between 6 and 7, with good chlorine dosage control, would minimize both NO_3^- and NCl_3. If good chlorine dosage control is not achieved, operation at pH 8 would minimize the noxious NCl_3 formation, giving a slightly increased NO_3^--N residual. A control scheme incorporating water flow, influent NH_3-N concentration, and effluent free chlorine is being developed at the EPA-DC pilot plant (15).

During the tests, the carbon columns removed all of the free residual chlorine. However, leakage of combined chlorine (0.3 to 0.9 mg./l. as Cl) occurred. Long term continuous operation is needed to evaluate the chlorination-carbon column system with respect to residual chlorine removal.

Chloride ion, a direct product of the reaction, was added to the product water as NaCl or $CaCl_2$. With an influent NH_3-N of 15 mg./l., 200 mg./l. of NaCl was produced.

Cost Estimates

The total operating cost developed by Bechtel Corporation for selective ion exchange within a 300 million gal./day physical-chemical treatment system was estimated at 9.7 cents per thousand gallons (Table 6). The estimate included the lime-salt method of regeneration, with sulfuric acid scrubbing of the ammonia-laden air. The acid scrubbing system, not yet piloted, would produce and require disposal of ammonium sulfate. The

clinoptilolite cost was estimated at 18 cents per pound and included mining and grinding to the 20 x 50 mesh particle. It was also assumed that waste heat from the sludge incinerator was used for heating the stripping air.

The cost of chlorination for a 300 million gal./day plant was estimated at 6.7 cents per thousand gallons (Table 7). For an inlet NH_3-N concentration of 15 mg./l., the chlorine was dosed at 120 mg./l., and lime usage was one pound CaO per pound of chlorine.

Conclusions

Within the physical-chemical treatment system, the ion exchange process, with clinoptilolite, was capable of continuously removing 95% of influent NH_3-N when the pilot facility was well manned. However, operation with minimum operator surveillance produced only 75% removal of the NH_3-N. Biological growth on the zeolite was not cumulative from run to run when using lime for regeneration.

Breakpoint chlorination of filtered secondary effluent in an agitated reactor removed essentially all the influent NH_3-N, with 95% conversion to nitrogen gas. The pilot plant study confirmed previous laboratory work (6) with chlorine dosages to obtain a Cl:N weight ratio between 8 and 9:1 and byproduct formation of NO_3^- and NCl_3. The byproducts NO_3^- and NCl_3 were minimized by good mixing, pH control, and proper chlorine dose rate. The conversion of NH_3-N to N_2 was incomplete with poor mixing at the chlorine application point.

The combination of selective ion exchange and breakpoint chlorination of the residual ammonia reduced the NH_3-N concentration to less than 0.1 mg./l.

With efficient ion exchange operation, byproduct formation of nitrate, nitrogen trichloride, and combined chlorine during breakpoint chlorination would be negligible because of low influent ammonia concentration.

Acknowledgements

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Nomenclature

BOD = 5-Day Biological Oxygen Demand, mg./l.

COD = Chemical Oxygen Demand, mg./l.

TKN = Total Kjeldahl Nitrogen, mg./l.

TOC = Total Organic Carbon, mg./l.

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Table 1. Selective ion exchange and residual pollutants

	COD mg./l.		TOTAL P mg./l.		SUSPENDED SOLIDS mg./l.	
	IN	OUT	IN	OUT	IN	OUT
March	53	42	.452	.505	3.8	7.1
April	54	41	.265	.164	12	6.4
May	44	38	.182	.160	5.5	21.0
June	42	38	.183	.100	3.4	5.1
July	30	26	.133	.080	5.0	9.0
August*	16	19	.101	.090	6.8	7.0
September	13	23	.112	.109	3.0	3.6
December	20	17	.092	.077	6.7	24.4

* Ion exchange placed after carbon adsorption

Table 2. Breakpoint chlorination of filtered secondary effluent

NH_3-N mg./l.			Cl:N	Free Cl
Influent	Effluent	pH	Wt. Ratio	mg./l.
15.4	<0.1	8.0	8.4:1	2.5
12.9	<0.1	7.0	9.0:1	3.4
13.9	<0.1	6.0	8.4:1	7.0
21.0	<0.1	7.0	8.5:1	2.0
17.0	<0.1	7.5	9.2:1	8.5
20.3	<0.1	6.0	8.8:1	6.5

Table 3. Caustic requirement in breakpoint chlorination

pH	$\frac{\text{pounds NaOH}}{\text{pound } Cl_2}$	Cl:N wt. ratio
6.0	0.88	8.8:1
7.0	1.3	9.0:1
7.9	1.67	9.0:1

Table 4. Nitrate formation

pH	NH_3-N in mg./l.	NO_3^--N mg./l.	% NO_3^--N of influent NH_3-N
6.0	13.9	0.5	3.6%
6.0	20.3	0.8	3.9%
7.0	17.0	0.9	5.3%
7.0	16.1	0.8	5.0%
7.9	20.3	1.2	5.9%
8.0	15.4	0.9	5.9%

Table 5. Nitrogen trichloride formation

pH	NH_3-N in mg./l.	Cl:N wt. ratio	NCl_3-N mg./l.
6.0	20.3	8.75:1	0.33
7.5	17.0	9.2:1	< 0.05
7.0	17.0	10.0:1	< 0.05
7.0	16.1	12.0:1	0.43
7.9	20.3	9.0:1	< 0.05

Table 6. Selective ion exchange costs*

	Cents
Electricity	0.5
Chemicals	2.6
Supplies	1.4
O & M Labor	0.6
Capital charges**	4.6
Total	9.7

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* Plant size 300 million gallons/day based on June 1970 costs, cents/thous. gallons.

** Annual capital cost computed at annual rate of 8% including interest and amortization.

Table 7. Breakpoint chlorination costs*

	Cents
Electricity (1 k.w.h.)	1.00
Chemicals**	
Chlorine (120 mg./l.)	3.75
Lime (1 lb./lb. Cl_2)	1.20
Supplies	.05
O & M Labor	.10
Capital charges***	.60
Total	6.70

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* Plant size 300 million gal./day, 15 mg./l. NH_3-N in, cents/thous. gallons

** Chlorine costs $75/ton; Lime costs $24/ton

*** Annual capital cost computed at annual rate of 8% including interest and amortization.

Figure 1. Selective ion exchange pilot plant.

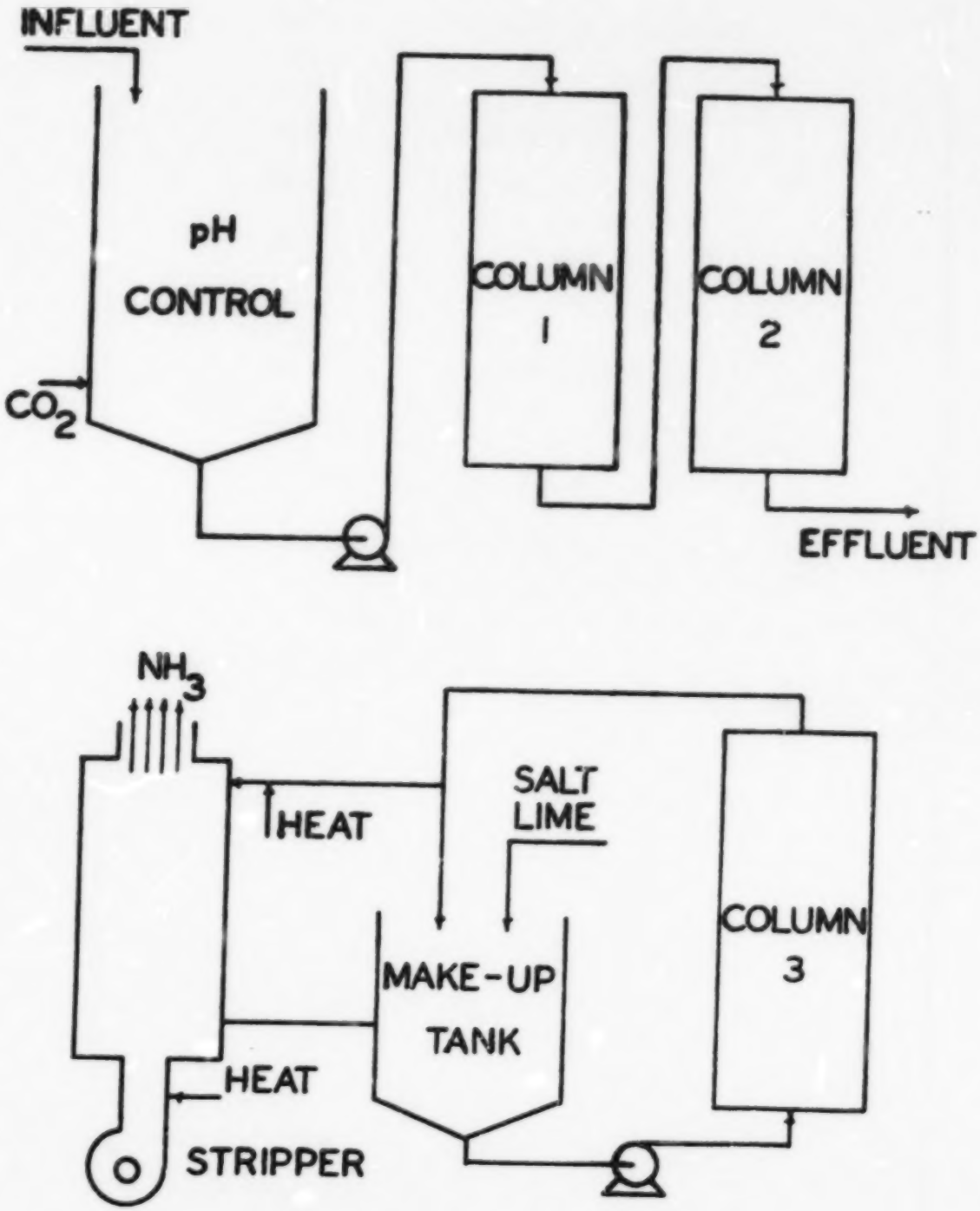
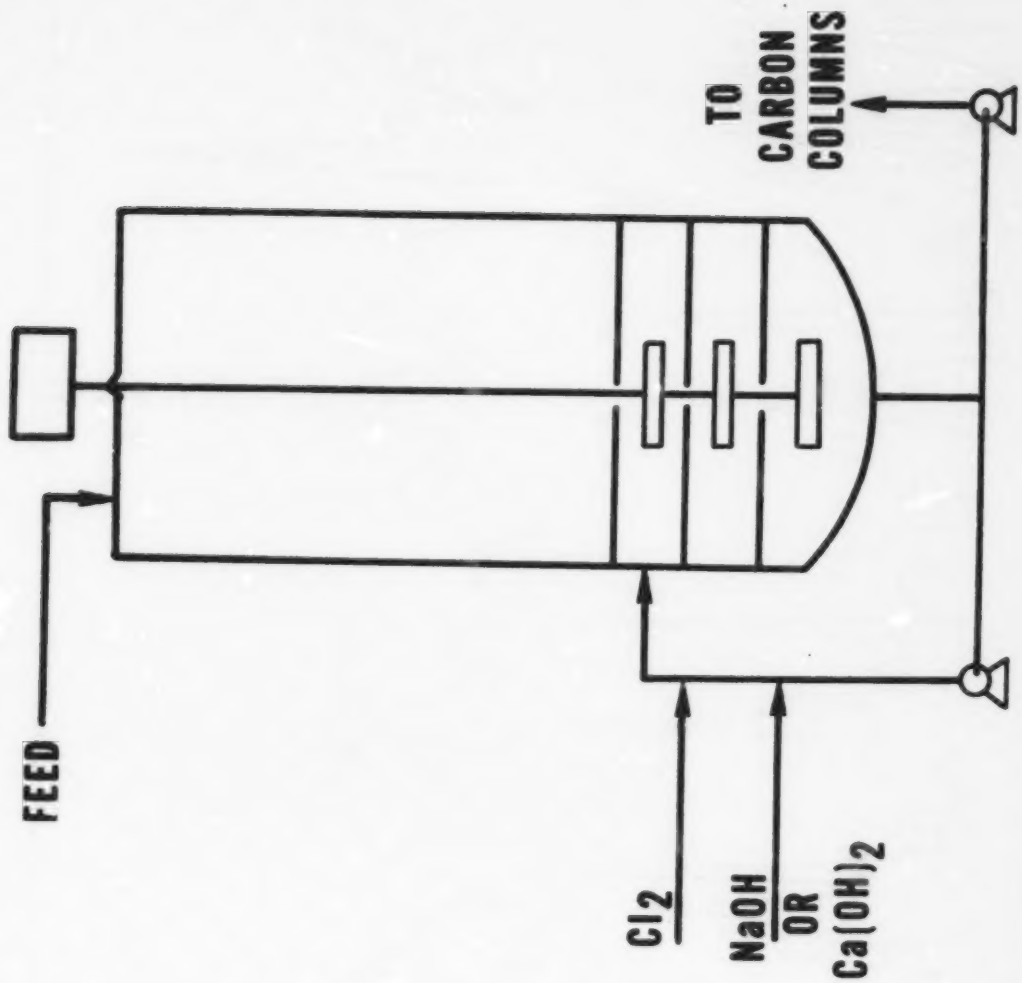


Figure 2. Breakpoint chlorination reactor



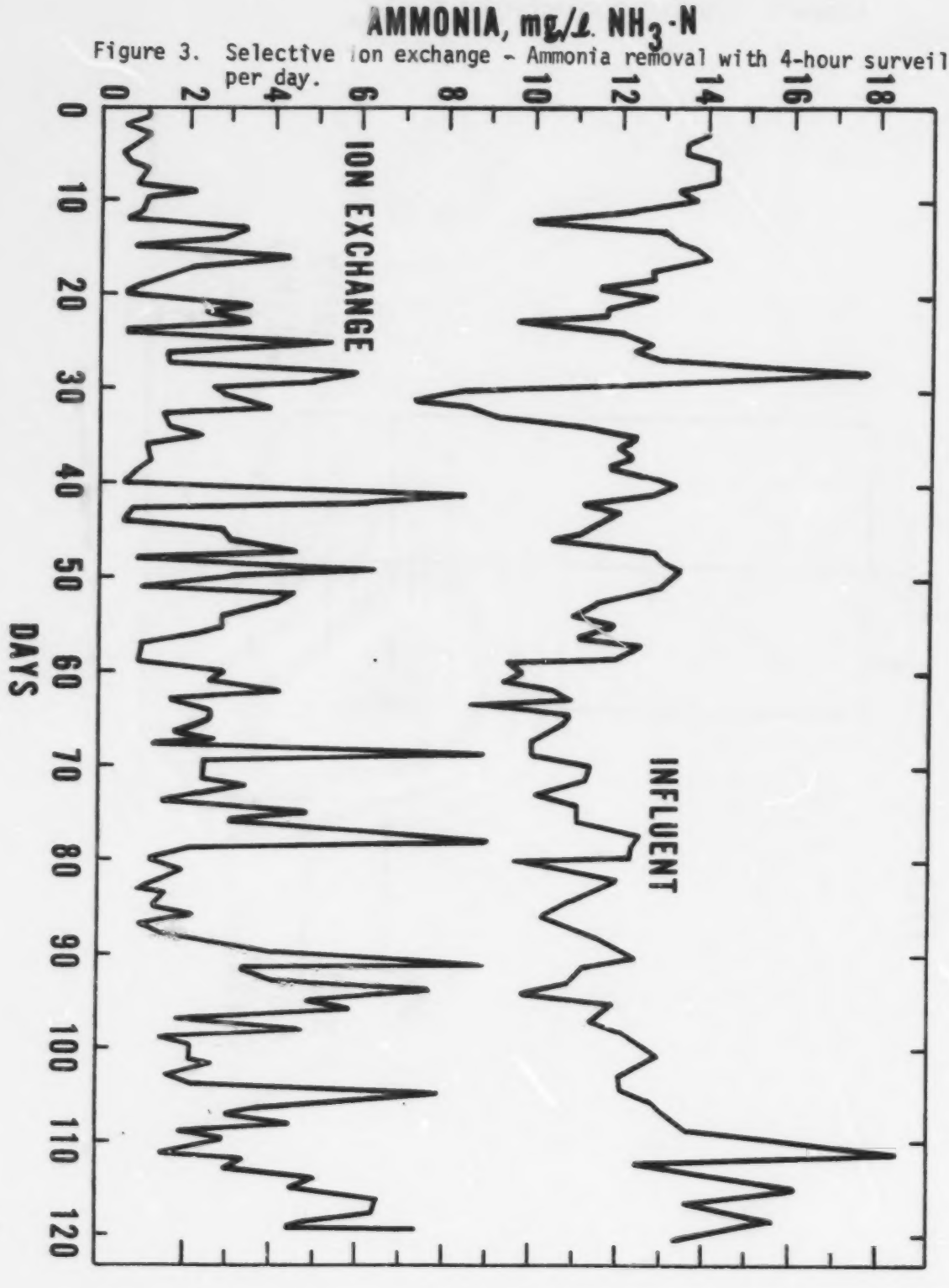


Figure 3. Selective ion exchange - Ammonia removal with 4-hour surveillance per day.

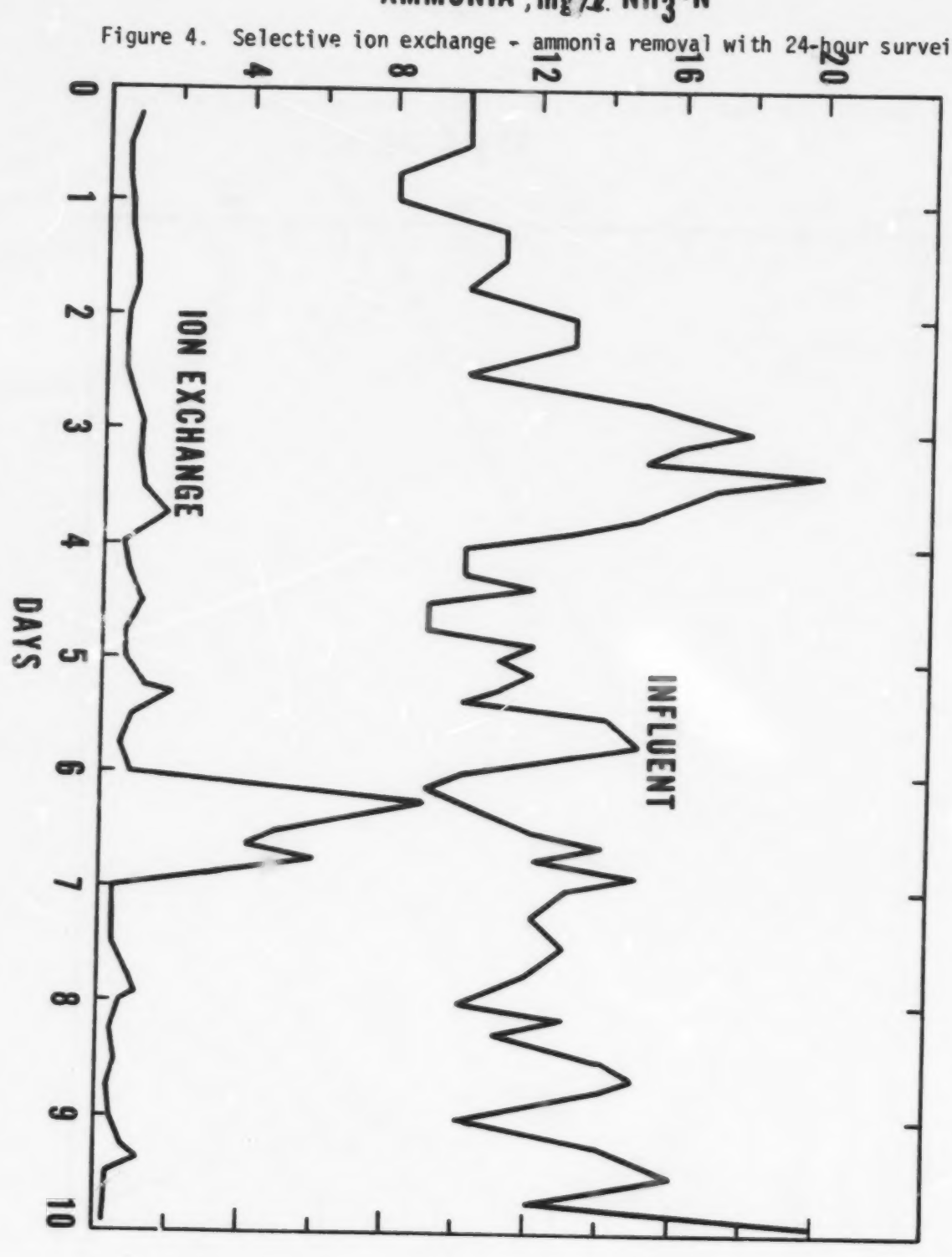


Figure 4. Selective ion exchange - ammonia removal with 24-hour surveillance.

Figure 5. Breakpoint chlorination of selective ion exchange effluent.

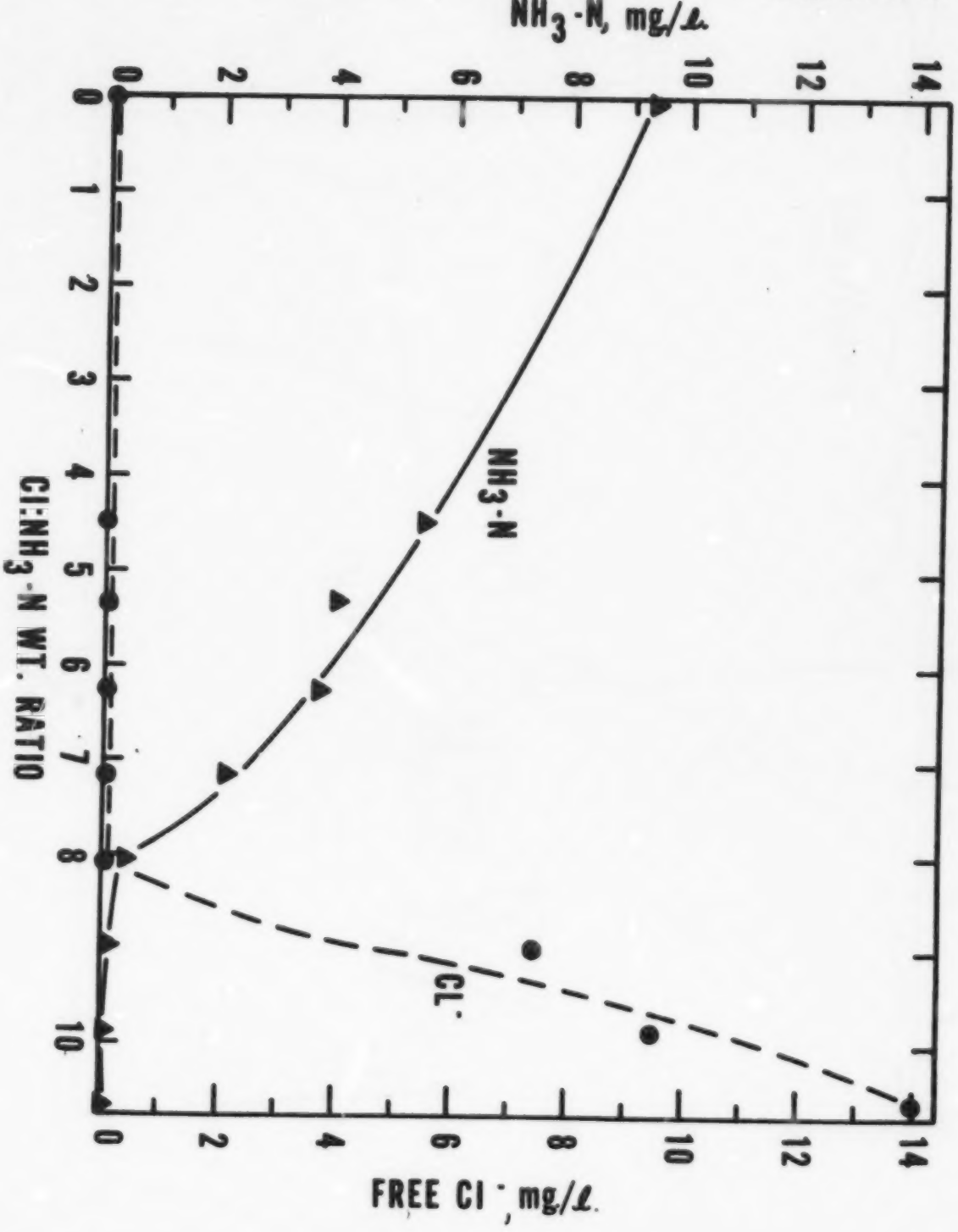


Figure 6. Breakpoint chlorination of filtered secondary effluent at pH 7.0-7.5.